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ODOUR POLLUTION - IDENTIFICATION
OF MALODOROUS ORGANIC COMPOUNDS LIBERATED DURING NATURAL
GAS PIPELINE TESTING OPERATIONS

ONTARIO MINISTRY OF THE ENVIRONMENT

During periodic maintenance operations, sections of natural gas pipelines are filled with millions of gallons of water for pressure testing. On discharge, this water was observed by ministry regional staff to be malodorous and discoloured. Laboratory tests indicated that settleable iron oxidehydroxide particles were responsible for the colour and that the odour was caused by volatile organic compounds. These organics, which were identified by gas chromatography - mass spectrometry, include the following compounds: dimethyl disulphide, methyl-ethyl-disulphide, diethyl disulphide, ethylbenzene, diethyl benzenes, toluene, diethyl toluenes, dimethyltetrahydronaphthalene and trimethyltetrahydronaphthalene. No mercaptans were found.

Fish placed in the wastewater showed no signs of distress after 48 hours exposure. Because of the low concentration (50-100 ng/l) (parts per trillion) of the relatively easily degraded organics and the apparent lack of toxicity of the wastewater to fish, along with the fact that this water is generally discharged in remote areas, the problem is regarded at present to be mainly of a nuisance or aesthetic character.

### MIREX IN ONTARIO

While a number of polychlorinated biphenyls (PCBs) and organochlorine pesticides have been regularly monitored during the past few years in Ontario, Mirex, another highly chlorinated organic insecticide, is an unexpected newcomer to the provincial environmental scene. Mirex, not registered for use in Ontario nor in adjacent states of the U.S., was first identified in Lake Ontario fish in 1974. More recently, it has been found in high concentrations in Lake Ontario herring gulls and has been the suspected cause of the reproductive failure observed in these herring gull colonies.

The gulls are voracious fish eaters and therefore are excellent indicator species for bio-accumulative trace contaminants in the aquatic environment. Since these contaminants also represent a potential danger to humans, MOE scientists, in December, 1975, decided to survey Great Lakes coho salmon and other sports fish for Mirex levels. Fish collections of the Ontario Ministry of Natural Resources were available for use in the project, however, some serious analytical problems were encountered.

Ideally, Mirex should be determined on the same sample, simultaneously with PCBs or other organochlorine pesticides using gas Copyright Provisions and Restrictions on Copying:

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chromatography with an electron capture detector. Unfortunately, all column packings commonly used for PCBs failed to separate Mirex.

An intensive search of the more sophisticated high temperature packings lead to an organoboron GC liquid phase ("Dexsil-300 GC") column packing which enabled Mirex to be separated from all significant PCB components. With this modified method, Mirex can be quantitated simultaneously with PCBs and the detection limit is 5 to 10 ppb. Recoveries from a variety of spiked environmental samples were above 80%.

Another important requirement in trace analysis is the positive identification of the target substance. Because results obtained in the identification of Mirex by electron impact mass spectrometry were considered ambiguous, chemical ionization mass spectrometry was applied. In isobutane ionizing media, strong molecular (M<sup>+</sup>) and (M-35)<sup>+</sup> (loss of a chlorine atom from Mirex) ions were obtained. These observations, taken together with gas chromatographic retention time data, confirmed the presence of Mirex in Lake Ontario coho salmon tissue.

#### PSEUDOMONAS AERUGINOSA

Members of the bacterial genus Pseudomonas can be found in industrial sites, hospitals and the environment, of which the most common human isolate is Ps. aeruginosa. This species is an opportunistic pathogen and is the predominant causative agent of eye and ear infections associated with the recreational use of water. A rapid primary isolation method for the enumeration and identification of Ps. aeruginosa from surface waters would therefore serve as a useful epidemiological tool in the investigation of health hazards in recreational waters.

One of the selective media designed for this purpose was the mPA medium of Levin and Cabelli (1972). During 1974 and 1975, the Toronto and London Laboratories evaluated this medium for its specificity and sensitivity. Over 424 colonies were isolated and subjected to 15 confirmatory tests. Of 393 positive colonies tested, 91.6% were in fact Ps. aeruginosa. From the typically negative colonies isolated,

94.0% belonged to other species and genera. These results corresponded well with the 92% accuracy and 95% specificity found by Levin and Cabelli.

As a result of this study, mPA medium has been adopted for routine enumeration of Ps. aeruginosa. It was shown that the utilization of acetamide, growth at 42°C, fluorescence, pigment and caseinase production in Skim Milk Agar, in addition to Gram stain and oxidase tests were sufficient confirmatory tests.

Since introducing the mPA procedure into surveys of the Great Lakes in 1974 and 1975, Ps. aeruginosa has been found to be directly associated with pollution sources and to decrease rapidly in concentration with increasing distance from the source. This parameter will be used to assess bathing water quality in specific surveys to widen the data base.

### STORM RUNOFF A SOURCE OF POLLUTION

A recent study, conducted jointly by Environment Ontario and the Canada Centre for Inland Waters, showed that storm water runoff contains high levels of pollution indicator bacteria and some pathogenic organisms. The runoff from streets is by no means pure rainwater, since its bacterial composition resembles dilute sewage and poses a health hazard. Fecal material from animals and birds washed from the streets during rain storms appears to be the source of pollution.

Among the pathogenic bacteria isolated, four species of <u>Salmonella</u> were consistently found in two different storm sewer systems. Since the members of the <u>Salmonella</u> group can cause various persistent intestinal disorders in man, their presence in storm water runoff represents a potential public health risk.

The results of this study showed the seriousness of storm water runoff as a factor in "non-point" source pollution of surface waters.

#### SOLUTION STANDARDS

The purchase of instrumentation to wash, fill and seal glass ampoules has

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allowed a new dimension to be introduced into the quality control program of the Laboratory Services Branch. Standard solutions of a wide variety of routine water quality parameters, sealed in these ampoules, can now be distributed on request to laboratories wishing to confirm the accuracy of their in-house standards. These ampoules are being used routinely in the Ministry central and regional laboratories to monitor for deterioration of working standard solutions whose stability may be affected by constant handling over a period of time.

Concentrated and diluted solutions to cover three different ranges of concentration have been prepared. The concentrates usually contain only one parameter and may be used to spike natural samples to check for matrix effects and other interferences, or to check calibration. The diluted solutions which are intended for direct analysis may contain several parameters (e.g. calcium, magnesium, sodium and potassium or ammonia, nitrite, nitrate and phosphate).

Specialty ampoules can be prepared for specific studies 14 For example, ampoules containing C were prepared for use in primary productivity studies, and another set was prepared for direct calibration of a CO<sub>2</sub> instrument.

### AUTOMATION OF CYANIDE ANALYSIS

For several years surveys carried out by the Ministry indicated cyanide levels in St. Mary's River well above the Ministry's maximum permissible concentration criterion of 0.2 mg/l. It has been shown that the highly toxic cyanide wastes originate from Algoma Steel Co, which daily discharges several hundred kilograms of cyanide into the river.

Such surveys, comprehensive enough to assess the extent of the pollution and to identify the source(s), require the analysis of large numbers of samples in a relatively short period of time. In the past, the laboratory was not always able to meet this requirement due to the time-consuming nature of the accepted analytical techniques and the instability of the cyanides. (Even properly preserved samples are stable for only up to one week).

The laboratory is now evaluating an automated procedure which will not only increase the capacity tenfold, but will lower the detection limit at the same time. Method modifications to allow the concurrent determination of thiocyanates, which are also present in steel mill effluents, are being investigated as well.

The evaluation centers around three procedures; the manual standard method, which is long and cumbersome but provides reliable results for most samples; automated procedure, which incorporates UV digestion and automatic distillation with a standard colorimetric measurement step, and the gas-liquid chromatographic procedure. The latter can determine free cyanide and is planned to be used in conjunction with a version of the automated procedure, in order to meet the requirements for the increasing number of cyanide tests for extended surveys. The new methodology will also be useful in monitoring the effectiveness of measures to prevent the discharge of cyanides or thiocyanates into waters such as the St. Mary's River, where cyanide may present an environmental health problem.

### PAH IN ONTARIO URBAN ATMOSPHERE

The presence of harzardous compounds in the atmosphere can often be traced to emissions from industry and other manmade sources. Some compounds of this nature belong to the group of polynuclear aromatic hydrocarbons (PAH) and have With carcinogenic properties. objective of establishing the occurrence and distribution of such compounds in urban and industrial atmospheres, a twoyear survey was conducted on two important members of this group: Benzo(a)pyrene (BaP), one of the most potent known, and benzo(k)fluocarcinogens ranthene (BkF), which is widespread and indicative of the presence of other PAH compounds formed during combustion processes of organic material.

Eleven urban communities in Ontario were surveyed and because of the large number of tests involved, existing methods required modification. These changes allowed the Laboratory to produce the information required for an assessment of PAH levels, and also helped to indicate their origin.

Two methods were used, both of which required the extraction of PAH's from air particulates, chromatographic separation of the individual components of the extracts on alumina, and by fluorescence measurement of the components.

This approach presented an analytical problem, because BkF always accompanies BaP and the two are difficult to separate. Since BkF is normally present at the same concentration as BaP, a complete separation is not usually necessary as they may be differentiated from two readings on the overlapping fluorescence curves. This is essentially the method routinely used. However, if the BaP concentration is more than 2.5 times the BkF level, the two must be separated in order to obtain reliable results.

Laboratory scientists developed a method based on complete resolution of the fluorometric emission curves by using specially activated alumina to chromatographically separate the two compounds. Both methods are simplified by passing the column effluent into a flow-through cell of a spectrophotofluorometer and measuring the area of the emission curve produced. Both methods are sensitive to about 0.01 ug BaP in 1000 m air.

The survey revealed that Welland had by far the highest level of PAH (up to 116 ug BaP/1000 m air) followed by Hamilton and Windsor. While levels for these areas seemed anomalously high in comparison to levels in cities of other countries, the PAH levels found in Toronto, Cornwall, Sarnia and Sudbury were found to be usually quite low (<1 ug BaP/1000 m air).

With the aim of establishing the underlying causes for the PAH levels observed, attempts were made to correlate these levels with a number of environmental conditions. In industrial areas, certain wind directions were found to coincide with peaks in pollution levels, which suggested or confirmed emission sources. Periods of temperature inversions over large areas of Ontario were invariably marked by surges in PAH levels of up to eight times the average levels. Incidences of high PAH levels were also caused by local events, such as fires, roof and road-tarring operations, and high traffic density. Correlations of the observed PAH levels with coefficient of haze values, with levels of airborne particulates and with annual and seasonal changes were examined for the communities surveyed.

#### METHYLMERCURY IN ONTARIO WATERS

The health concerns regarding the consumption of methylmercury contaminated fish have resulted in a considerable research effort on several aspects of mercury in the aquatic environment. One such area is the route for methylmercury accumulation in fish. It has been established experimentally that mercury uptake from food as well as directly from water are both important.

Established analytical techniques are capable of determining methylmercury levels in fish and sediment, but to date, there was no satisfactory method which could have been routinely applied for reliable analysis of natural waters.

The major analytical problems stem from the extremely low concentrations of methylmercury and/or mercury ions usually encountered in natural waters. These concentrations are generally below the detection limit of the conventional FAAS methods for total mercury in water (0.05 ug/l).

Several methods for the determination of methylmercury in water have been published, but to date the only one capable of detecting natural (background) levels of methylmercury was the method developed by Chau and Saitoh (CCIW). Using this method as a starting point, a technique for the analysis of methylmercury in natural waters with concentrations as low as 0.5 ng/l (parts per trillion) has been developed The method at the MOE laboratories. consists of a continuous liquid-liquid extraction of a four litre acidified water sample with benzene. The benzene layer is back extracted four times with an aqueous The cystein-methyl-1-cystein solution. mercury complex is then broken with hydrochloric acid, and the resulting methylmercuric chloride is re-extracted into benzene. This cystein back extraction is then repeated and the combined 2 ml benzene extract thus obtained is analyzed for methylmercuric chloride by electron capture gas chromatography. Spiking studies have indicated that the method recovers 80-85% of the methylmercury at the 2 ng/l level.

The procedure as it stands is fairly time-consuming, so its application to large numbers of samples is not yet feasible. However, several sets of samples from the mercury-contaminated Wabigoon River

have been analyzed by this method. The preliminary results indicate that the method might be useful in identifying those regions of the river system where active methylation of mercury by sediment microorganisms is occurring.

Results obtained on methylmercury concentrations in water would help establish the relative importance of two routes of uptake on the accumulation of mercury in fish. The results may also provide an indication of recovery times as a measure of the effectiveness of restoration procedures for contaminated areas.

### ARSENIC DETERMINATION IN MOIRA RIVER SAMPLES

The Moira River meanders through southeastern Ontario, finally entering Lake Ontario at Belleville. It is primarily a recreational system with many cottages along its banks. Unfortunately, at Deloro, the upper Moira River flows through an industrial site which although long abandoned, is still emitting very high levels of arsenic to the river system. Since 1880 the Deloro property has been the site of a gold and silver mine and arsenic and cobalt smelters. The property is littered with slag heaps and disposal pits. One disposal area extends over 7 acres and contains primarily calcium arsenite, while another 17 acres is almost totally ferric arsenate.

There is a rudimentary collection and precipitation system which treats the runoff prior to discharge to the river. The collection system is obviously inadequate. The Ministry of the Environment laboratories, in analyzing routine samples obtained by southeast region personnel, have found levels of over 4 mg/l arsenic in the river at points of ground water ingress. These concentrations are lowered by non-contaminated inflows to yield an approximate mean level of 0.2 mg/l downstream and 0.1 mg/l in Moira Lake. The maximum allowable concentration for arsenic in water supplies is 0.05 mg/l.

The toxicity of arsenic is well established, and arsenic is presently the only environmental pollutant known to have directly caused human deaths in Canada. Although these cases occurred several decades ago near the Moira River, they provide graphic examples of the need to continuously monitor drinking water for

suspected contaminants. Arsenic, especially in its airborne elemental form, is also suspected to be a carcinogen. In water, the form of arsenic is of paramount importance as arsenites are approximately twenty times more toxic than arsenates. Therefore, in order to establish the extent of the problem, the exact source(s) and the most effective means of treatment, it is necessary to determine the form as well as the total amount of arsenic entering the river.

Laboratory staff are using an automated procedure based on the generation of arsine gas by borohydride reduction from an acid digested sample. The arsine is sparged into a heated quartz cell where it is converted to atomic arsenic which is measured by conventional atomic absorption spectroscopy. Under normal running conditions the system has a detection limit of 0.5 ug/l with a between run relative standard deviation of 4%, on river samples containing .01 to .03 mg/l As. accuracy of the method was confirmed by analyzing several standard reference materials.

The acid digestion oxidizes all forms of arsenic to the arsenate ion. In this process very volatile organoarsenicals such as trimethylarsine are lost; however, these compounds are rarely found in natural waters. The method is therefore suitable for the determination of total non-volatile arsenic.

A combined polarographic and automated buffered hydride generation analysis system for arsenate, arsenite and organoarsenic speciation is also being investigated.

### DETERMINATION OF CARBON IN AIRBORNE DUST

Carbon, in its elemental or free form is an annoying and visible air pollutant. It is associated with soiling of surfaces, especially in urban areas from sources such as automobile and diesel exhaust, incinerators, heating, power generation and manufacturing operations. It may also be a carrier of hazardous pollutants which could be adsorbed on its surface.

As data on carbon levels in ambient air are few, a method has been developed for the chemical analysis of free and total carbon in suspended air particulate matter collected on glass fibre filters in a high volume sampler. Free carbon includes graphite, coal, coke, carbon black, soot, etc. Total carbon includes the carbon content of organic fibres, carbonates, biological materials, etc., in addition to free carbon.

In experiments to determine free carbon, various reagents were tested. Some were found to attack free carbon while others would not dissolve unwanted materials. Concentrated nitric acid, used under controlled conditions, was chosen as the best compromise.

Since some forms of free carbon may be of extremely fine particle size, the method of filtration is critical. In addition, the filter medium must be inorganic and unaffected by concentrated nitric acid. A novel method of filtration was developed for this purpose. The carbon-containing suspension is filtered in a gooch crucible through a glass fibre filter treated with aluminum and titanium oxides. This was found to be an excellent filter for carbon, retaining even carbon black. After filtration, the contents of the gooch crucible are transferred quantitatively to the combustion crucible of a carbon analyzer, where the carbon is burnt in an induction furnace in a stream of The resulting carbon dioxide is oxygen. of measured bν means a conductivity detector. Interferences are removed by adsorption on selective filters.

Total carbon is determined by directly combusting a dried aliquot of the high-volume air filter in the carbon analyzer.

The limit of detection of the method, based upon a filter aliquot, 6.25 cm<sup>2</sup>, corresponds to approximately 1.5 ug of carbon/m of air. The precision of the method is - 10% as determined by analyzing aliquots of the same air filter.

Using these procedures, free and total carbon levels have been determined in the air of some urban centres in Ontario. Typical values found for free carbon and total carbon range from 5 to 30 ug/m<sup>3</sup>, and 10 to 50 ug/m<sup>3</sup> of air respectively.

# ANALYSIS OF SELENIUM IN ENVIRONMENTAL SAMPLES

The effect of selenium on human and animal health is a topic of growing importance. Once condemned as a toxic element and a cancer-causing agent, selenium is

emerging as a necessary and beneficial element in human and animal nutrition. Recent demographic studies indicate an inverse relationship between selenium content of people's diet and the incidence of cancers of internal organs.

Selenium is also believed to reduce the toxicity of methylmercury in some organisms, and has been demonstrated to accumulate in a stoichiometric relationship with methylmercury for a few species.

Because of the importance of selenium in the environment and in biological processes, considerable effort has been spent on developing reliable and sensitive methods suitable for large-scale use.

A comparison was made between the following methods:

- fluorometric diaminobenzidine (DAB)
- fluorometric diaminonaphthalene (DAN)
- hydride generation/flameless atomic adsorption spectrophotometry (FAAS)
- gas chromatography using the pheniline diamine complex of Se (GC). The results obtained are tabulated below.

Method	Detection Limit ug/l	Tests/Day	
DAB	10	30	
DAN	0.1	20	
FAAS	1.0	40	
GC	0.5	20	

FAAS method was chosen for routine determinations because of ease of operation and speed. The virtually interference-free GC method has comparable sensitivity and although more time-consuming, it is a reliable alternative to the FAAS procedure.

In search of increased sensitivity, the laboratory also developed an electro-analytical method using differential pulse cathodic stripping voltammetry (DPCSV). This method is an order of magnitude more sensitive than those above (DL = 0.05 ug/l), and is simple and fast (25 tests/day). Samples are acid digested, the pH is adjusted to 1.4 - 0.1 and the selenium is codeposited with added Cu onto a hanging mercury drop electrode. The intermetallic Se/Cu deposit is electro-reduced. The resulting cathodic current is proportional to the amount of Se present in the

digestate and provides a measure of the Se concentration in the sample.

Analyses of USBS standard reference materials using the FAAS and DPCSV methods produced excellent recoveries for both methods

CONCENTRATION *		
STATED	FAAS	<b>DPCSV</b>
1.1 + 0.1	1.2	1.27
0.08 + 0.01	0.07	0.07
5.0	4.9	4.9
16.0	15.4	16.9
48.0	45.3	47.8
	1.1 ± 0.1 0.08 ± 0.01 5.0 16.0	STATED FAAS  1.1 ± 0.1 1.2  0.08 ± 0.01 0.07  5.0 4.9  16.0 15.4

<sup>\*</sup> USBS material concentrations are in ug/g EPA waters are in ug/l.

The increased sensitivity of these methods and the ability to corroborate Se analyses will allow a thorough documentation to be made of the level of this important element in the waters, fish and sediments of the Province.

### ST. LAWRENCE CEMENT COKE BURN STUDY

With the ever increasing demand for safe and efficient disposal methods for toxic waste materials, the St. Lawrence Cement Company has embarked on a study program, in cooperation with this Ministry and Environment Canada. The study involves the use of by-product coke, the residue from distillation of recovered crude oil from Alberta tar sands, as a fuel in a rotary cement kiln.

Both "dry" and "wet" procedures are used in the preparation of the kiln feed. In the "dry" process, the raw materials are initially ground in a dry state and are then further dried as required. In the "wet" process, the raw materials are ground with water to produce a slurry. The finely

ground and precisely compounded "dry" feed or "wet" slurry is then burnt to Portland cement in the kiln. The kiln types used in the study are steel cylinders, slightly inclined from the horizontal, varying from 150 to 500 feet in length and 8 to 16 feet in diameter, with a speed of rotation of approximately one r.p.m. Fuel in the form of powdered coal, coke, oil or gas is blown into the lower end of the kiln. The raw feed is introduced at a controlled This mode of rate at the upper end. operation produces a high-temperature burning zone near the discharge end. In operation, experimental fuels containing varying amounts of the by-product coke are burnt in the kilns.

Tar sands coke contains sulphur, nickel and vanadium as impurities. The fate of these elements within the kiln is determined by stack emission analysis and process material balance. Stack sampling is being carried out by the Ontario Research Foundation, with the Environment Ontario laboratories participating in the analytical part of the study. As the amounts of particulate material obtained for analysis on trial bases were limited, it was found necessary to employ a carbon rod flameless atomic absorption technique for determination of stack sample metal content.

The kiln fuel burning process should, with suitable modifications, lead to methods of disposal of potentially toxic wastes such as chlorinated hydrocarbons, including P.C.B. A report on the study is to be issued in the near future.

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ANALYSIS Editorial Committee

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#### FROM THE PAST

Alterow do uniarad

NNUAL EXAMINATIONS: 1859.

FIRST YEAR

### ELEMENTARY CHEMISTRY.

Examiner: HENRY CROFT, D.C.L.

- 1. What is the meaning of chemical affinity?
- 2. What is meant by double decomposition? Give instances.
- 3. What peculiarities are observed during the union of chemical substances.
- 4. In what respect does chemical attraction differ from all other kinds of attraction?
  - 5. What is meant by the specific gravity of a body?
  - .6. How may the specific gravity of a body be ascertained?
- \*7. What is meant by the boiling point of liquid, and what circumstances cause it to vary?
- 8. Describe fully the preparation and properties of hydrogen.
- 9. What is the percentage composition of water, and how is this ascertained?
- \*10. What impurities usually exist in water, and how may they be detected?
- \*11. Explain the nomenclature of the acids, exides, and salts, and give the proper names for the following substances: I'c'O', S'O', SO', SO', MnO, SnO', KO.CO', KO. 2 GrO:

This exam paper from 1859 was found in 1971 under the floorboards when the "Soldiers Tower" was renovated.

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sei, & preparation and properties of olefat Bas.

From we urces are the salts of potassium ob med?

15. What is a mon salt, and how is it obtained? What is the action of sulphurle acid upon it?

- 16. Give an account of the most important compounds of iron?
- . \*17. Mention the proximate elements of plants and their formulæ.
- \*18. What is the composition of alcohol, whence obtained, and how acted on by oxygen?

\*Honor Questions.

Question 10 is indeed an honour question, 118 years later we are still working on the answers.

## RECENT PAPERS AND REPORTS PREPARED BY LABORATORY STAFF

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- R. D. Smillie, T. Sakuma and W. K. Duholke. Low Molecular Weight Aromatic Hydrocarbons in Drinking Water. Presented by R. D. Smillie at the Air and Water Quality Symposia at the Third Annual Meeting of the Federation of Anatytical Chemistry and Spectroscopy Societies in November, 1976, in Philadelphia.

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- G. S. Hendry and P. Bolton. <u>Bacteriological</u>
  Water Quality of Round <u>Lake</u>, <u>Belmont</u>
  Twp.
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